DMR

Dinnel Marine Resources

QUALITY ASSURANCE EVALUATIONS OF AMPHIPOD BIOASSAYS OF UPPER COLUMBIA RIVER SEDIMENTS

Final Report

8 November 2005

For

CH2M Hill
Bellevue, Washington

Prepared By

Dinnel Marine Resources
Anacortes, WA

DMR-0507

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Table of Contents

List of Tables

Appendices

1.0 INTRODUCTION

The bioassay laboratory selected to conduct amphipod testing of Upper Columbia River sediments was Northwestern Aquatic Sciences (NAS), Newport, Oregon. NAS conducted two 28-day amphipod (*Hyalella azteca*) bioassays of the Columbia River sediment samples. NAS is a State of Washington accredited laboratory (Lab accreditation number C042, expiration: 30 September 2005) and is certified to perform amphipod bioassays of sediments. A copy of NAS's accreditation certificate and Scope of Accreditation appears in Appendix 1.

This report summarizes the Quality Assurance/Quality Control (QA/QC) evaluations of two freshwater amphipod (*Hyalella azteca*) survival and growth tests conducted by NAS. Fifty sediment samples, plus reference and control sediments, were tested by NAS during two batches of testing.

The QA steps taken to ensure high quality data and maximum data completeness before, during and after testing are described in this report. Major QA tasks included the following:

- A pre-test review of NAS's *Hyalella* protocol
- An audit of the first of the two bioassays while testing was in progress
- An initial evaluation of all data for completeness, correct data entries, and correct calculations
- A final QA evaluation of overall data quality and usability (this report).

2.0 QUALITY ASSURANCE AUDIT RESULTS

2.1 REVIEW OF THE LABORATORY PROTOCOL

NAS's protocol for the 28-day *Hyalella* test was reviewed by Dinnel Marine Resources (DMR) prior to initiation of testing. The freshwater amphipod test using *Hyalella azteca* as the test animal was based on two published protocols: 1) ASTM Standard Method E1706-00 (ASTM 2001) and 2) EPA Test Method 100.1 (EPA 2000). Following review of NAS' test protocol (NAS-XXX-HA4c, Revision 3), DMR discussed several minor clarifications via phone with NAS and NAS's test protocol was accepted.

2.2 TEST-IN-PROGRESS AUDIT

Because of the large number of sediment samples to be tested, the sediment bioassays were conducted in two batches. The first amphipod bioassay began on 28 April 2005 and the second bioassay was started on 5 May 2005.

Dr. Paul Dinnel, DMR, preformed one test-in-progress audit at NAS's laboratory in Newport, OR on 3 May 2005 when the first batch of sediments was being tested. All testing procedures closely followed NAS's approved protocol and all test parameters appeared to be within testing guidelines. A checklist for the audit performed on 3 May 2005 appears in Appendix 2.

2.3 INITIAL DATA EVALUATIONS

All raw data forms and electronic database files were reviewed for completeness and fidelity of transcription to electronic formats. A 100% check was made of all data entered into NAS's internal electronic database and spot checks were made of all Excel spreadsheet calculations and formulae. All errors, omissions, clarifications, or changes needed were documented and communicated to NAS. Copies of DMR's Quality Assurance Data Evaluation Comments to NAS and NAS's response letter appear in Appendix 3.

2.4 FINAL QA EVALUATION OF OVERALL DATA QUALITY AND USABILITY

Following corrections to the data reports by NAS personnel, a 100% check of the corrections was made on 2 November 2005 to verify each correction. All corrections made by NAS were deemed satisfactory. Following this, an overall evaluation of data completeness and quality was accomplished. DMR's conclusions regarding data completeness and quality follow below (summary details for these tests are given in Tables 1and 2).

2.4.1 Chain of Custody and Sample Holding

All chain of custody protocols were properly observed in transfers of sediment samples from CH2M Hill to NAS. The test sediments were stored at 4° C in the dark until tested.

2.4.2 Amphipod, *Hyalella azteca,* Sediment Bioassay No. 727-1

1. A *Hyalella azteca* bioassay was conducted on 25 Upper Columbia River sediment samples, plus 6 reference sediments. A negative control sediment, and one positive (toxic) control test with cadmium was run concurrently with the Upper Columbia River test sediments.

2. Testing was initiated within 8 days following sediment collection.

3. This test was completed with no protocol or water quality deviations.

4. The reference toxicant 50% Lethal Concentration (LC50) for the cadmium test was 7.89 µg/liter. This result was within NAS's control chart warning limits of 3.26 – 10.7 µg Cd/liter.

5. Negative control mean mortality (3.7 %) was <20% for this test and thus acceptable by present ASTM and EPA criteria. The mean control dry weight was 0.41 mg/individual.

6. The maximum ammonia concentration measured in the overlying water during this test in any one sample was 1.1 mg/liter total ammonia. This is well below the known toxicity threshold of 20 mg/liter total ammonia (4-day LC50 -- Ankley et al. 1995 as cited in ASTM 2001).

7. Sulfides were not measured during this testing program.

8. Replication was eight-fold for all samples.

9. Data completeness for the 25 (plus 6 reference and 1 control) samples tested with *Hyalella* was essentially 100 %.

10. **Final QA determination:** All data are of excellent quality and fully usable for any purpose.

Table 1. Summary of *Hyalella* Test 727-1.

Hyalella azteca, 28 April – 26 May 2005:

Number of test samples, including reference sediments: 31
Sediment holding time <2 weeks?: Yes
Protocol deviations?: No
Average negative control mortality: 3.7 %
Range in reference sediment mortality: 0.0-5.0 %
Reference toxicant LC50: 7.89 µg/liter cadmium. This value is within NAS's control chart warning limits.
Water quality parameter deviations: None
Ammonia concentrations < critical limits?: Yes

QA reviewer conclusion: All data are of excellent quality and fully usable for any purpose.

2.4.3 Amphipod, *Hyalella azteca,* Sediment Bioassay No. 727-2

1. A *Hyalella azteca* bioassay was conducted on 25 Upper Columbia River sediment samples, plus 6 reference sediments. A negative control sediment, and one positive (toxic) control test with cadmium was run concurrently with the Upper Columbia River test sediments.

2. Testing was initiated within 13 days following sediment collection.

3. This test was completed with no protocol or water quality deviations.

4. The reference toxicant 50% Lethal Concentration (LC50) for the cadmium test was 4.40 µg/liter. This result was within NAS's control chart warning limits of 3.34 – 10.8 µg Cd/liter.

5. Negative control mean mortality (2.5 %) was <20% for this test and thus acceptable by present ASTM and EPA criteria. The mean control dry weight was 0.38 mg/individual.

6. The maximum ammonia concentration measured in the overlying water during this test in any one sample was 0.9 mg/liter total ammonia. This is well below the known toxicity threshold of 20 mg/liter total ammonia (4-day LC50 -- Ankley et al. 1995 as cited in ASTM 2001).

7. Sulfides were not measured during this testing program.

8. Replication was eight-fold for all samples.

9. Data completeness for the 25 (plus 6 reference and 1 control) samples tested with *Hyalella* was essentially 100 %.

10. **Final QA determination:** All data are of excellent quality and fully usable for any purpose.

Table 2. Summary of *Hyalella* Test 727-2.

Hyalella azteca, 5 May – 2 June 2005:

Number of test samples, including reference sediments: 31
Sediment holding time <2 weeks?: Yes
Protocol deviations?: No
Average negative control mortality: 2.5 %
Range in reference sediment mortality: 2.5-6.3 %
Reference toxicant LC50: 4.40 µg/liter cadmium. This value is within NAS's control chart warning limits.
Water quality parameter deviations: None
Ammonia concentrations < critical limits?: Yes

QA reviewer conclusion: All data are of excellent quality and fully usable for any purpose.

3.0 REFERENCES

Ankley, G.T., M.K. Schubauer-Berigan and P.D. Monson. 1995. Influence of pH and hardness on the toxicity of ammonia to the amphipod *Hyalella azteca*. Can. J. Fish. Aquatic Sci. 52:2078-2083.

ASTM (American Society for Testing and Materials). 2001. Standard test methods for measuring the toxicity of sediment-associated contaminants with fresh water invertebrates. ASTM Standard Method No. E1706-00. Am. Soc. Test. Materials, West Conshohocken, PA.

EPA (U. S. Environmental Protection Agency). 2000. Section 11, Test Method 100.1, *Hyalella azteca* 10-d survival test for sediments. Pp. 47-54 in: Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, Second Edition. EPA/600/R-99/064.

Appendix 1

Northwestern Aquatic Sciences' State of Washington Accreditation Certificate and Scope of Accreditation

The State of Washington
Department of Ecology

This is to certify that

Northwestern Aquatic Sciences
Newport, OR

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective October 1, 2004, and shall expire September 30, 2005.

Witnessed under my hand on October 7, 2004.

Perry F. Brake, Chemist
Lab Accreditation Section Manager

Lab Accreditation Number
C042

Scope of Accreditation

Northwestern Aquatic Sciences

Newport, OR

is accredited by the State of Washington Department of Ecology to perform analyses for the parameters listed below using the analytical methods indicated. This Scope of Accreditation may apply to any of the following matrix types: non-potable water, drinking water, solid and chemical materials, and air and emissions. Accreditation for all parameters is final unless indicated otherwise in a note. Accreditation is for the latest version of a method unless otherwise specified in a note. EPA refers to the U.S. Environmental Protection Agency. SM refers to American Public Health Association's publication, Standard Methods for the Examination of Water and Wastewater, 18th, 19th or 20th Edition, unless otherwise noted. ASTM stands for the American Society for Testing and Materials. PSEP stands for Puget Sound Estuary Program. Other references are detailed in the notes section.

Matrix Type/Parameter Name	Reference	Method Number	Notes
Non-potable Water			
Ampelisca abdita	ASTM	E 1367	1
Ampelisca abdita	EPA	100.4	13
Ampelisca abdita	PSEP	1995	2
Atherinops affinis (West Coast)	EPA	1006.0	3,4
Bioaccumulation, Benthic Invert	ASTM	E 1688	5
Bioconcentration, Fish, Mollusks	ASTM	E 1022	6
Ceriodaphnia dubia	EPA	1002.0	4,7
Ceriodaphnia dubia	EPA	2002.0	4,8
Chironomus tentans	EPA	100.5	14
Chironomus tentans	ASTM	E 1706	9
Chironomus tentans	EPA	100.2	14
Crassostrea gigas	PSEP	1995	2
Crassostrea gigas (West Coast)	EPA	1005.0	3,4
Cyprinodon variegatus	EPA	1004.0	4,10
Cyprinodon variegatus	EPA	2004.0	4,8
Dangerous Waste Static Salmonid	WDOE	80-12 Part A	11
Daphnia magna	EPA	2021.0	4,8
Daphnia pulex	EPA	2021.0	4,8

Matrix Type/Parameter Name	Reference	Method Number	Notes
Dendraster excentricus	ASTM	E 1563	12
Dendraster excentricus	PSEP	1995	2
Dendraster excentricus (West Coast)	EPA	1008.0	3,4
Eohaustorius estuarius	ASTM	E 1367	1
Eohaustorius estuarius	PSEP	1995	2
Eohaustorius estuarius	EPA	100.4	13
Holmesimysis costata	EPA	821-R-02-012	4,8
Holmesimysis costata (West Coast)	EPA	1007.0	3,4
Hyalella azteca	EPA	100.1	14
Hyalella azteca	ASTM	E 1706	9
Hyalella azteca	EPA	100.4	14
Leptocheirus plumulosus	ASTM	E 1367	1
Leptocheirus plumulosus	EPA	100.4	13
Menidia beryllina	EPA	1006.0	4,10
Menidia spp.	EPA	2006.0	4,8
Mysidopsis bahia	EPA	1007.0	4,10
Mysidopsis bahia	EPA	2007.0	8
Mytilus spp.	PSEP	1995	2
Mytilus spp. (West Coast)	EPA	1005.0	3,4
Neanthes arenaceodentata	PSEP	1995	2
Salvelinus fontinalis	EPA	2019.0	4,8
Oncorhynchus mykiss	EPA	2019.0	4,8
Pimephales promelas, Chronic	EPA	1000.0	4,7
Pimephales promelas	EPA	2000.0	4,8
Rhepoxynius abronius	ASTM	E 1367	1
Rhepoxynius abronius	PSEP	1995	2
Rhepoxynius abronius	EPA	100.4	13
Strongylocentrotus purpuratus	ASTM	E 1563	12
Strongylocentrotus purpuratus (WC)	EPA	1008.0	3,4
Strongylocentrotus purpuratus (WC)	EPA	600/R-95/136	3,4

Matrix Type/Parameter Name	Reference	Method Number	Notes
Strongylocentrotus spp.	PSEP	1995	2

Accredited Parameter Note Detail

(1) ASTM. "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," E 1367-99. (2) Puget Sound Estuary Program, "Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediments," July 1995. (3) USEPA. "Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms," EPA 600/R-95/136 (Third edition) August 1995. (4) Meets requirements of "Laboratory Guidance and Whole Effluent Toxicity Test Review Criteria," Washington Department of Ecology, Publication Number WQ-R-80, Revised December 2001. (5) ASTM. "Standard Guide for Determination of the Bioaccumulation of Sediment Associated Contaminants by Benthic Invertebrates," E 1688-00a. (6) ASTM. "Practice for Conducting Bioconcentration Tests with Fishes and Saltwater Bivalve Mollusks," E 1022-94. (7) USEPA. "Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms," EPA-821-R-02-013 (Fourth Edition) October 2002. (8) USEPA. "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms," EPA-821-R-02-012 (Fifth Edition) October 2002. (9) ASTM. "Test Method for Measuring the Toxicity of Sediment-associated Contaminants with Freshwater Invertebrates," E 1706-00. (10) USEPA. "Short-term Methods for Measuring the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms," EPA-821-R-02-014 (Fourth Edition) October 2002. (11) Washington Department of Ecology. "Biological Testing Methods," WDOE 80-12 Revised April 1997. (12) ASTM. "Guide for Conducting Static Acute Toxicity Tests with Echinoid Embryos," E 1563-98. (13) USEPA. "Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods." EPA 600/R-94/025 June 1994. (14) USEPA. "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates." EPA 600/R-99/064 (Second Edition) March 2000.

Authentication Signature

Perry Brake – Section Manager, Washington State Department of Ecology – Lab Accreditation Section

Appendix 2

Results of the Test-in-Progress Audit by Dinnel Marine Resources

Dinnel Marine Research

CHECKLIST FOR ~~10~~ 28-DAY FRESHWATER AMPHIPOD BIOASSAY

Project Name: UPPER COLUMBIA RIVER

Auditor: PAUL DINNEL

Laboratory: NORTHWESTERN AQUATIC SCIENCES, NEWPORT, OR.

Test Type: 28 DAY SURVIVAL & GROWTH

Test Personnel: GERALD IRISSARRI, GARY BUHLER, SUSAN GAGE, BAILY PRIDGEON.

Test SOP: NAS-XXX-HA4C

Test Date: 4/28/05

Number of Samples: 25 TEST, + 6 REFS, + 1 CONTROL

SOP Deviations: NO

Other Notes:

Shipping and Holding Conditions

Samples Received: 31 SAMPLES

Samples Tested: 25 TEST + 6 REF. + 1 CONTROL

Holding Time at Test Initiation: 8 DAYS

Holding Conditions: 4°C IN DARK

Problems Noted in Shipping and Holding: NO

Testing Conditions

Protocol Used: NAS-XXX-HA4C

Protocol Available?: YES

Deviations?: NO

Test Initiation Date: 28 APRIL '05

Number of Samples: 31

Multiple Batches?: YES (2)

Test Species: HYALELLA AZTECA

Animal Source: CHESAPEAKE CULTURES

Holding Conditions: 20°C, HARDNESS REDUCED FROM 170 TO 100 mg/l

Holding Time: 2 DAYS

Feeding During Holding?:

Size Selection Criteria: 7-8 DAYS OLD

Other Notes:

Test Chamber Size: 300 ml GLASS BEAKERS

CHECKLIST FOR 10-DAY FRESHWATER AMPHIPOD BIOASSAY

Quality Assurance Audit

Audit date: 3 May '05

Days/Hours After Initiation: 5 Days

Source of Neg. Control Sediment: Beaver Creek, OR

# of Reference Sediments: 6

Amount of Sediment Used: 100ml

Final Water Volume: 275 ml

Freshwater Source: Modified tap water Dechlorinated

Freshwater Treatment: Hardening reagents added

Freshwater Holding Time: 3 Days

Number of Replicates: 8

Sediment Equilibration Period?: 24 Hours

Beakers/amphipods Randomized?: YES

No. amphipods/Beaker: 10

Feeding During Test: 1 ml YTC/Day/Beaker

Test Chambers Aerated?: No

Water Temperature: 22.0-23.9°C

Water Hardness: 68-128 mg/l

Water DO: 3.2-8.1 mg/l

Water pH: 6.9-7.7

Photoperiod: 16 Light : 8 Dark

Positive Controls Used?: YES

Positive Control Toxicant: Cd

Daily Test Records Maintained?: YES

Emergence Data Collected?: No

QA Officer: Linda Nemeth

Internal QA Checks?: Not Yet

Reburial Measured at End of Test? No

Sulfides and Ammonia Measured at Initiation and End?: Ammonia in overlying water at beginning and end.

SOP Deviations or Problems Noted: None

QA Officer: Paul A. Dinnel

Audit Date: 3 May '05

Appendix 3

Comments by Dinnel Marine Resources to Northwestern Aquatic Sciences Following DMR's QA Review and NAS's Response Letter

DMR

Dinnel Marine Resources
1519 13th St.
Anacortes, WA 98221
360-299-8468

23 October 2005

Mr. Gerald Irissarri
Upper Columbia River Bioassay Project Manager
Northwestern Aquatic Sciences
PO Box 1437
Newport, OR 97365

Dear Gerald:

I have finished my audits of your two draft data reports for the testing of Upper Columbia River test sediments. Your data reports were in excellent condition and reflect your usual high degree of attention to detail. There are only a few minor "housekeeping" items that need some attention and a couple of transcription errors that need to be corrected for your final drafts. My audit findings are noted below. Please provide me with copies of any corrections made to your draft data reports.

Both Draft Reports

1. You might want to take this occasion to update your references to the ASTM and EPA protocols noted on the top of page 1 of your "Toxicity Test Report." Your NAS protocol references ASTM 2001 and EPA 2000. The most recent ASTM Standard is 2005 (E 1706-05). I'm not sure what the most recent EPA reference is.

2. In a number of places in your reports you use the term "biomass." In this case you are actually measuring the "dry weights" or "growth" of *Hyalella* and not biomass. Biomass as defined by ASTM Designation E 1705-95 (reapproved in 2002) is "total weight of living matter in a given volume." I suggest changing "biomass" to "dry weight" or "growth" as appropriate. Places where I noted the term "biomass" in your reports include: bottom of page 2, top of page 3 (twice), top of page 4, Table 2 and in formula in the header for the "Endpoints Data Entry and Calculations File."

3. On page 4 of both reports you note that mean control growth of ≥0.15 mg is required. This requirement is neither in your protocol nor in any of the ASTM or EPA protocol editions that I have. Is this a project-specific requirement? (Note: ASTM and EPA protocols state that there should be "measurable growth of test organisms in control sediment.")

4. Tables 3 and 4: You note that the "+"s in the table indicate responses that are *significantly different from* the reference sediments. My review suggests that in each of these cases the +s denote responses that are significantly *higher* (mortality) or *lower* (growth). If this is the case, it would probably help the project sponsor and the subsequent reviewers to realize that you mean one-tailed responses instead of two-tailed responses.

5. Under "Miscellaneous Notes" (bottom of page following "Sediment Descriptions" page), the value to convert ft-candles to lux (0.0929) is crossed out and a corrected value of 0.929 is inserted. Actually, the original value (0.0929) is the correct value.

6. I note that on one Chain of Custody sheet the "cooler temperature" and "custody seal intact?" information is missing. Do you have any information in any other logs to add this information to these sheets?

Report for Test No. 727-1

1. Endpoints Data Entry and Calculations File: It appears from my photocopy that the tare weight for beaker 108 (index 235) should be 29.268 instead of 29.265. This is a very minor difference, but a correction should be made, if necessary, and the sample means, etc. recalculated.

Report for Test No. 727-2

1. Endpoints Data Entry and Calculations File: Dry weight for beaker 23 (index 51) should be 33.687 instead of 33.678. Please correct and recalculate sample means, etc.

2. At the end of your Excel WQ sheet, you record a mean and SD for ammonia. There actually are no means or SDs since many of the values are < values.

3. ToxCalc sheet, page 5 of 7: The sample type in the upper right corner is copper sulfate, whereas it should be cadmium chloride.

Once again, the corrections suggested above are all relatively minor. Both your sediment testing and draft reports appear to be in excellent shape.

Sincerely,

Paul A. Dinnel
Quality Assurance Auditor

CC: Frank Dillon, CH2M Hill

NORTHWESTERN AQUATIC SCIENCES
A Division of NAS Associates, Inc.
P.O. Box 1437, Newport, Oregon 97365 • (541) 265-7225 • Fax: (541) 265-2799 • contact@nwaquatic.com

October 27, 2005

Dr. Paul Dinnel
Dinnel Marine Research
1519 13th Street
Anacortes, WA 98221

Dear Paul:

Enclosed are corrections in response to your QA audit of reports P727-1 and P727-2.

In regards to your statement about updating the references in our protocols, we hope to do that soon. However, these tests were conducted using our protocols at the time, which are based upon ASTM Method E 1706-00 ASTM 2001 and EPA/600R-99/064 (Which I believe is the most current edition.). If there is a change to ASTM 2005 with regard to the *Hyalella* test, our protocols would not have reflected the change and subsequently the tests would not have incorporated the change. So, we believe it's necessary to leave in the old references.

If you have any questions, please do not hesitate to call me at 541-265-7225 or email girissarri@nwaquatic.com.

Sincerely,

Gerald Irissarri
Project Manager

Encl.